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PATENT SPECIFICATION.

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COMPLETE SPECIFICATION

Plastic Compositions and methods of making same.

We, THE TITANIUM ALLOY MANUFACTURING COMPANY, a corporation organized and existing under the laws of the State of Maine, United States of America, of 111, Broadway, City and State of New York, United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to improved compositions, which are capable of being formed into sheets or films and are especially useful in electric light reflectors, and to methods of making the same.

In many industrial fields the use of plastics is rapidly expanding and replacing other materials, such as glass. However, in one particular field in which glass is used progress has been slow due to a peculiar combination of desired properties which has been difficult to obtain in plastics. This field comprises electric lamp shades and reflectors for use in the home and industry.

A desirable property in a light reflector material is the property of reflecting the maximum amount of yellow light possible and also of transmitting the maximum amount of blue light possible. Thus it has been found that working efficiency and general comfort, as regards eye strain, are increased when a maximum of yellow light is reflected and only a diffuse blue light is transmitted through the reflector for illumination of the room proper.

The object of the present invention therefore is to provide a composition which can be formed into a sheet or film having the property of reflecting yellow light and transmitting blue light.

According to the invention, the composition is made by dispersing in a transparent or translucent base material consisting of a synthetic plastic, a percentage, which is inversely proportional to the thickness of the sheet or film to be formed and is between 0.03 and 0.4% by volume for a sheet or film, one millimeter in thickness, of a white pigment in the form of particles of a substantially uniform size between 0.4 and 0.6 micron and of isometric, tetragonal, orthorhombic or hexagonal crystal form and having a refractive index substantially different from that of the base material.

Suitable synthetic plastics for use in forming the compositions of the present invention are translucent or transparent synthetic resins of a phenolic, acrylic, styrene, poly-styrene or urea-formaldehyde base, and synthetic materials capable of being formed into sheets or films and having a basis of a cellulose derivative, for example, cellulose nitrate, cellulose acetate and cellulose xanthate.

White pigments which may be incorporated in such synthetic materials and which meet the required conditions physically and may be prepared in the proper range of particle size include cerium dioxide, tin oxide, zirconium dioxide, titanium dioxide, thorium dioxide, antimony oxide, columbium oxide, tantalum oxide and various co-precipitated or complementary precipitated products of these substances with other materials such as barium sulphate, calcium sulphate, zinc oxide, lithopone etc.

Various suitably fired mixtures or combinations of the above substances may also be used, for example a fired combination of zinc oxide, titanium oxide and antimony oxide has the proper physical properties and can be prepared to have the proper particle size. A suitably fired combination of titanium oxide, antimony oxide and fluospar may also be used.

The preparation of each material will, of course, have to be modified slightly in accordance with the nature of the particular material which is used.

To illustrate the invention, zirconium dioxide of uniform particle size 0.5 micron, white in colour, almost completely tetragonal or orthorhombic and having a high refractive index has been prepared in the following manner: A substantially pure zirconium dioxide is first converted to the sulphate which is dissolved in water to form a saturated solution. Any soluble iron present is reduced by nascent hydro-

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gen, or zinc metal, or sodium thiosulphate, etc., or by so regulating the original sulphating of the zirconium dioxide that an insufficient amount of acid is present to combine with all the oxide so that all the iron is present as ferrous sulphate. A soda ash solution is added to the properly diluted zirconium sulphate solution (which had been previously clarified), until the precipitate forming no longer dissolves. A solution of potassium sulphate is then added in the ratio of 20 to 50 mols. of zirconium dioxide to 1 mol. of potassium sulphate, and the addition of soda ash is continued until the solution reaches a pH of 3. to 4., so as to hold back the precipitation of ferrous iron which occurs at $pH=5.5$. The precipitate is then filtered and washed free of iron and sulphates. The cake is dried and calcined at 1600 to 1800° F. until all acid fumes are eliminated; the process usually requires 8 to 12 hours.

The resulting calcine is suitably disintegrated, and about 6 parts of soda ash is intimately mixed with about 100 parts of the cold calcine. This mixture is calcined at 1660° to 1700° F., preferably as close to 1680° F. as possible, for a period of time sufficient to produce the proper crystallization as determined by periodic microscopic examinations.

The course of the last reaction is as follows: Pronounced peptization takes place at first, thereby forming very minute crystals of a size just at or below the limit of resolution of the highest power of the microscope viz. less than 0.2 micron. These crystals serve as nuclei of crystal growth, which continues at an astonishingly uniform rate. The change is then removed from the furnace as soon as these nuclei grow to 0.5 micron in size. If the reaction is allowed to continue, the size of the crystals will continue to increase. On such large particles it can be easily seen that the particles are almost entirely tetragonal or orthorhombic in shape. Normally about 1½ hours is required to complete the proper growth at 1680° F.

The calcine obtained is then cooled, and to every 100 pounds of such calcine is added 15 pounds of 1.16 sp. gr. hydrochloric acid. Water is then added to produce a stirring slurry and the mixture is digested for 12 hours at room temperature. It is then washed twice by settling and decantation, the water is replaced and the pH adjusted to between 5. and 6. with ammonia water. The mixture is then filtered and the cake is washed free of chlorides and thereafter disintegrated in the slurry in a ball mill, and cleaned by passing through a fine lawn. The resulting product is then dried.

Properly sized zirconium dioxide of suitable characteristics may also be prepared in many different ways, providing the particle size of zirconium dioxides so prepared is finely regulated by the method hereinbefore described, viz.: the controlled calcination of the zirconium dioxide with soda ash.

The compositions obtained were tested in the following manner. Varying amounts of this 0.5 micron particle size zirconium dioxide of tetragonal or orthorhombic form were incorporated in a urea formaldehyde resin, for example, by mixing the pigment in the solid resin and then ball-milling same for a proper length of time, by trituration, or simply by mixing if the particles of the resin are sufficiently fine. Such mixture is usually obtained by grinding. Incorporation of the pigment in a liquid resin may be accomplished by ball-mill grinding, paint-mill grinding, simple mixing, trituration etc. The proper moulding is obtained in the case of a thermoplastic resin by the application of the proper temperature and pressure for a definite length of time which is standard procedure for forming the particular resin. The specific details vary from resin type to resin type, and are well-known. Paint or lacquer films are applied by pouring, dipping, brushing, or spraying, and are either allowed to dry at normal temperatures or are baked for specified times and temperatures to porduce the desired finished properties. Thermosetting resins are prepared in final form by heating in a mould for definite times and temperatures and then cooled.

In the case of zirconium dioxide and a base material 1 mm. thick, the selective light effects described were just noticeable at 0.03% addition by volume, and were very strong at 0.4% addition. The effects were rapidly lost at higher additions and became more and more non-selective at amounts above 0.4% by volume. As stated previously the percentage of the zirconium dioxide or other pigment to be added varies inversely as the thickness.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is :—

1. The method of making a composition capable of being formed into a sheet or film having the property of reflecting yellow light and transmitting blue light, which comprises dispersing in a transparent or translucent base material consisting of a synthetic plastic a percentage, which is inversely proportional to the thickness of the sheet or film to be formed

and is between 0.03 and 0.4% by volume for a sheet or film one millimeter in thickness, of a white pigment in the form of particles of a substantially uniform size between 0.4 and 0.6 micron and of isometric, tetragonal, orthorhombic or hexagonal crystal form and having a refractive index substantially different from that of the base material.

2. A method in accordance with Claim 1 in which the pigment is white zirconium dioxide.

3. A method in accordance with Claim 2 in which the zirconium dioxide is obtained by reacting a mixture of zirconium dioxide and soda ash at a temperature between 1660° and 1770° F. until the zirconia grains peptise to form as nuclei uniform particles of zirconia less than 0.2 micron in size, which are then allowed to grow to the required size.

4. The method of making a composition having the property of reflecting yellow light and transmitting blue light substantially as described.

5. A composition having the property of reflecting yellow light and transmitting blue light prepared by a method claimed in any one of the preceding claims.

6. A sheet material having the property of reflecting yellow light and transmitting blue light which comprises a dispersion in a transparent or translucent base material consisting of a synthetic plastic of a percentage, which is inversely proportional to the thickness of the sheet and is between 0.03 and 0.4% by volume for a sheet 1 mm. in thickness, of a white pigment in the form of particles of a substantially uniform size between 0.4 and 0.6 micron and of isometric, tetragonal, orthorhombic or hexagonal crystal form and having the refractive index substantially different from that of the base material.

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